

AUTOMATED FIBER PLACEMENT OF PEEK/IM7 COMPOSITES WITH FILM INTERLEAF LAYERS¹

A. Bruce Hulcher ², William I. Banks III, R. Byron Pipes and Surendra N. Tiwari
Old Dominion University, Norfolk, VA

Roberto J. Cano and Norman J. Johnston
NASA Langley Research Center, Hampton, VA

ABSTRACT

The incorporation of thin discrete layers of resin between plies (interleafing) has been shown to improve fatigue and impact properties of structural composite materials. Furthermore, interleafing could be used to increase the barrier properties of composites used as structural materials for cryogenic propellant storage. In this work, robotic heated-head tape placement of PEEK/IM7 composites containing a PEEK polymer film interleaf was investigated. These experiments were carried out at the NASA Langley Research Center automated fiber placement facility. Using the robotic equipment, an optimal fabrication process was developed for the composite without the interleaf. Preliminary interleaf processing trials indicated that a two-stage process was necessary; the film had to be tacked to the partially-placed laminate then fully melted in a separate operation. Screening experiments determined the relative influence of the various robotic process variables on the peel strength of the film-composite interface.

Optimization studies were performed in which peel specimens were fabricated at various compaction loads and roller temperatures at each of three film melt processing rates. The resulting data were fitted with quadratic response surfaces. Additional specimens were fabricated at placement parameters predicted by the response surface models to yield high peel strength in an attempt to gage the accuracy of the predicted response and assess the repeatability of the process. The overall results indicate that quality PEEK/IM7 laminates having film interleaves can be successfully and repeatably fabricated by heated head automated fiber placement [1].

Key Words: Thermoplastics, Fiber placement, Interleaving, Barrier films

¹ This paper is declared a work of the U.S. Government and is not subject to copyright protection in the United States.

² Currently employed at the NASA George C. Marshall Space Flight Center, Huntsville, AL, and to whom all correspondence should be addressed.

1.0 INTRODUCTION

The enhancement of the mechanical properties of composite materials through various methods has been investigated in some detail over the past several years. Most of these methods have focused on improving damage tolerance, interlaminar fracture toughness, and fatigue life. Design approaches for optimization of composite material mechanical properties include laminate stacking sequence, fiber orientation, z-axis reinforcement, modifications to the matrix resin, and hybrid laminate concepts including resin and metal interleaves [2].

The most important material property governing the fatigue behavior of composite materials is the toughness of the matrix resin component. Most composite material failures occur as the result of ply delamination, especially in mechanical fatigue. Attention has been directed toward the toughening of this inter-ply region. This may be achieved by modification of the matrix resin or by the incorporation of thin discrete layers of tough, ductile resin between plies. The addition of such interleaves has been shown to improve fatigue and impact properties by increasing the interlaminar fracture toughness. Ideal materials for use as interleaves are tough, high strain-to-failure thermoplastics that reduce shear stress concentrations at the ply interfaces.

The current study attempts to determine the feasibility of fabricating composite structure having film interleaf layers by heated-head fiber placement technology. Preliminary process development was carried out on the composite without the interleaf layer prior to conducting trials on the fabrication of interleaved laminates. A summary of the study and suggestions for future work are given.

2.0 EXPERIMENTAL

2.1 General

2.1.1 Fiber Placement Facility The NASA Langley Research Center automated fiber placement facility consists of an Asea Brown Boveri³ (ABB) Model IRB 6000, six-axis, fully-articulating robot [4]. Attached to this platform is the robot heated-head placement end-effector, designed and fabricated by Automated Dynamics Corporation. A fully-programmable rotating spindle is included which provides a seventh axis for fabrication of cylindrical components. The facility is also equipped with a heated flat tool for fabrication of open-section panels. The head consists of a tape dispensing system that feeds material from the spool creel, through a guide chute, and onto the placement surface. The head is capable of placement of up to five 0.635 cm wide composite ribbons or one 3.17 cm wide composite tape. A combination of hot gas and radiant heat sources are available to preheat both the incoming and substrate materials prior to laminate compaction and consolidation [4].

The hot gas source consists of two nitrogen torches that are capable of heating the gas to 900°C. The heated gas is directed into the nip-point region during placement via a single nozzle shared by the torches. A 1000 W tungsten halogen lamp serves as the radiant heat source [5]. The preheating sources may be used either individually or in tandem during processing depending on the thermal properties of the composite and other placement parameters.

The incoming tape is fed beneath a compaction roller that may be heated to 500°C by an internal cartridge resistance heater. Gas torch and compaction roller temperature control are accomplished by closed-loop feedback control systems. Roller temperature is monitored by an IR sensor located aft of the roller. Torch temperature is monitored by two thermocouples that are exposed to the gas stream within the shared gas exit nozzle. Laminate consolidation is effected by a pneumatically-actuated compaction roller which is capable of producing consolidation loads up to 1.47 kN.

2.1.2 Material The composite material used in the study was supplied by Cytec Fiberite³ and was manufactured by their proprietary 'TIFF' process. This material consisted of PEEK resin (T_g :145°C, T_m :342°C) and IM7 fibers in a fully-consolidated and dimensionally accurate tape form. The width and thickness of the as-received tape were 3.17 cm and 0.014 cm, respectively. Tape void content was determined by both acid digestion (ASTM D3171-76) and optical image analysis. Results from digestions indicate a void content of less than 1%. Optical image analysis of void content was performed on an Olympus BH-2 laboratory microscope using the Olympus Cue 2 image acquisition and analysis system. Thirty screen images totaling 0.06 cm² of area were analyzed. The mean void content as determined by image analysis was found to be 0.45%. A photomicrograph of five samples of the as-received composite tape is shown in Figure 1(a). The micrograph reveals smooth, flat ribbon surfaces on the well-consolidated tape form.

2.2 Summary of Composite Process Development Prior to initiating process development with interleaf films, optimization of the process for fabricating non-interleaved laminates was conducted. Unidirectional four-ply wedge peel specimens were fabricated and tested and the results, together with void content data, were used as process quality indicators [6]. Preliminary processing trials were conducted to determine the approximate ranges of hot gas torch temperature and compaction roller load that should be more fully investigated in a process optimization study.

A Box-Wilson designed experiment was chosen for process optimization. Thirteen experiments were conducted with roller temperature and compaction load ranges of 600°C - 800°C and 0.67 kN - 1.20 kN, respectively. Held constant were IR lamp output (100%), placement speed (2.54 cm/s), and compaction roller temperature (475°C). Based upon both peak and average peel strength results, the optimal settings for the compaction load and torch temperature were found to be 1.33 kN and 700°C. Void content by optical image analysis was obtained for each of the thirteen experiments and was found to range from 0.18% to 3.72%. Analysis of the void content and strength data revealed no statistical correlation between the two sets; final optimization was based solely upon the peel strength data [1]. A photomicrograph of a cross-section of a four-ply peel specimen (0.18%voids) is shown in Figure 1(b).

2.3 Interleaf Film Process Development

2.3.1 Preliminary Processing Trials Initial placement trials were conducted with 0.076 cm PEEK film. Single strips of composite tape 45.7 cm long and 3.17 cm wide were placed onto the tool surface at the conditions deemed optimal in the composite process development

³ The use of trademarks or names of manufacturers in this report is for accurate reporting and does not constitute an official endorsement, either expressed or implied, of such products or manufacturers by the NASA.

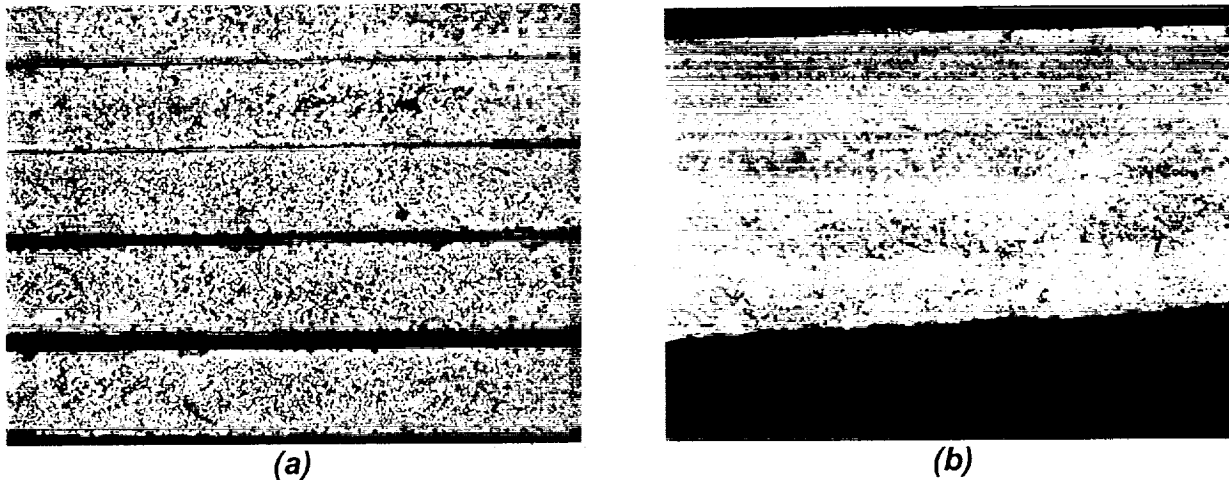


Figure 1. (a) Photomicrograph of as-received PEEK/IM7 composite tape. **(b)** Photomicrograph of cross-section of 4-ply composite peel specimen.

portion of the study. Strips of film were taped to the beginning end of the composite ply and draped along their length. These strips were manually constrained and placed in minimal tension at the free end during processing. This served to prevent the film from buckling upward from the substrate composite ply surface and thus from contacting the heated roller prior to the moment of adhesion.

An initial set of experiments was conducted using the heated compaction roller as the sole heat source. The roller compaction force and placement rate were held constant at 0.44 kN and 2.54 cm/s. The roller temperature was varied from 270°C to 520°C. This range was used such that actual film temperatures from above T_g to above T_m could be attained at the fixed placement speed of 2.54 cm/s. The results for roller temperatures of from 410°C to 420°C indicate that the films could be made to adhere lightly, or to be 'tacked', to the composite ply surface. The best results for light adhesion as indicated from visual inspection were obtained with roller temperatures of between 320°C and 400°C. These films were observed to be of high integrity and uniformity along the specimen length and could be readily peeled from the ply surface. This was an indication that neither the film nor composite matrix resin had fully melted and thus interfacial healing of the two components had not been achieved: measurement of the film thickness and width indicated no change of dimension, a fact that supported this conclusion.

A series of 4-ply peel specimens was fabricated to determine the peel strengths at the film/composite interface using only a film 'tack' processing stage. Although it was determined that the film had not fully bonded to the substrate ply after the tack pass, it was thought that placement of the subsequent composite ply might effect intimate healing of the film to both the lower and upper composite plies. Roller temperatures for the tack stage were varied between 300°C and 390°C. The compaction force during film processing was held constant at 0.56 kN. The composite plies were again processed at the conditions deemed optimal in the earlier composite processing experiments.

No general trend was found between the strength data and increases in roller temperature, indicating that film tack temperatures in this range do not significantly influence the quality of the interfacial bond. Although several of the peak strength values were high, the average strength values are well below those found previously for the composite alone. Visual inspection of the fracture surfaces after testing revealed resin-rich regions on the upper ply surfaces and bare composite surfaces on the lower plies. This would indicate significant adhesive failure of the film at the lower composite ply surface. Composite cohesive failure was observed as evidenced by fiber pullout, however was considered to be minimal for this specimen group. These results led to the conclusion that a second processing pass would have to be executed in order to more fully melt and bond the film to the lower composite ply surface.

A second set of peel specimens was fabricated in an attempt to increase film adhesion to the composite substrate ply. Films were lightly tacked to the substrate as previously accomplished and a second film 'melt' processing pass was performed. These melt staging trials were conducted at higher compaction roller temperatures and with additional thermal energy supplied by the IR radiant heat source. Earlier melt stage trials using a rotating compaction roller resulted in severe film de-bonding from the lower composite ply as the result of film pull-up and adhesion to the roller surface. This difficulty was effectively eliminated by constraining roller rotation during melt stage processing. The use of a 'sliding' roller was continued throughout the remainder of the study during the film melt processing pass.

Only marginal increases in peel strength were achieved for these initial tack/melt processing trials. The averages of both the peak and average peel strengths from the specimens having no melt stage were 9.34 kN/m and 3.62 kN/m, respectively. The averages for the results with melt stage processing for the same data were 9.64 kN/m and 4.25 kN/m, respectively. The fracture surfaces were visually inspected and adhesive failure of the film at the upper surface of the substrate composite ply was again observed, though to a lesser extent than for the previous experiment set. These results indicate that the use of a second processing pass is beneficial in terms of increasing film adhesion to the substrate ply.

A final set of preliminary experiments using the gas torches was conducted in an attempt to achieve complete film/composite adhesion. The first, second, and fourth composite plies were fabricated at the same processing conditions determined to be optimal in the composite process development portion of the study. Due to the possible influence of the processing parameters of the ply immediately preceding the film layer on specimen peel strength, the compaction load and torch temperatures for this ply were varied in the experimental set. Held constant for the film processing passes were the film tack stage roller temperature (340°C), the film melt stage roller temperature (480°C), the compaction load (0.56 kN) during both film tack and melt stage processing, and the lamp percent output during the melt processing stage (100%). Processing parameters that were varied for the experiments were the melt stage processing rate, the torch temperature during film melt staging and the 'upper' composite ply compaction load and torch temperature.

The results of the study are presented below in Table 1. The data represent a 43% increase in average peel strength and an 18% increase in peak peel strength as compared to the previous experiment set. In contrast to the previous studies, examination of the peel surfaces revealed the

absence of a distinct film layer on the upper ply fracture surfaces, indicative of an increase in adhesion of the film to the lower composite ply. Additionally, inspection of both lower and upper specimen fracture surfaces revealed fiber pullout and thus cohesive failure within the composite plies.

Table 1. Results of 4-ply peel testing of specimens processed at increased melt stage and upper composite ply processing temperature and compaction force. All composite plies placed at 2.54 cm/s. Note: Torch temperatures and loads listed for composite are for the 3rd, or upper, composite ply only.

Specimen Number	Load Composite (kN)	Torch Temp. Melt/Composite (°C)	Speed @ Melt (cm/s)	Peak Strength (kN/m)	Ave. Strength (kN/m)
1	1.33	- / 700	1.27	10.73	7.84
2	1.33	700 / 700	1.27	12.18	8.19
3	1.11	- / 700	1.27	11.94	4.89
4	1.11	- / 850	1.27	12.34	9.92
5	1.11	700 / 850	2.54	10.93	7.79
6	1.11	- / 850	2.54	11.42	8.33

Several observations regarding the results of these trials may be made. Specimens 1 and 3 of Table 1 were placed at the same conditions with the exception of the upper-ply composite compaction load. This force was 1.33 kN for specimen 1 and 1.11 kN for specimen 3. The average peel strength for specimen 1 (7.84 kN/m) was 38% higher than for specimen 3 (4.89 kN/m). The mean value of the average strength data of the specimens fabricated with a 700°C torch during upper composite ply processing was found to be 20% lower than the mean of those fabricated with a torch temperature of 850°C. Each of these findings would indicate that the processing parameters used during placement of the third composite ply are a significant factor in the process. The specimens having the highest average peel strengths overall, specimens 4 and 6, were processed without the gas torches during the melt stage. The only difference in the processing of these two specimens was the processing rate during the film melt stage. Specimen 4 was placed at half the rate of specimen 6 and shows an increase in both peak, and average peel strength. This would be expected from placement at lower rates; the amount of thermal energy available is a function of both thermal energy source temperatures and placement processing rates. It was also noted that three out of the top four specimens in terms of average peel strength were fabricated without gas torches during the film melt stage. This could be due to stagnation flow of the hot gas in the nip-point region; only small increases in actual material pre-heat temperatures result from relatively large changes in torch temperature. The results of the three preliminary film processing trials are presented in Table 2.

Table 2. Results of preliminary film processing trials.

Processing Method	Peak Peel Strength (kN/m)	Average Peel Strength (kN/m)
Tack Stage Only	9.35	3.62
Melt and Tack I	9.64	4.25
Melt and Tack II	11.59	7.83

2.3.2 Screening Experiments A relatively large number of process variables could possibly influence the adhesion quality and the resultant peel strengths of the film specimens. In an attempt to determine which, if any, of these variables have little or no measurable impact on the quality of the film-composite bond, a Plackett-Burman screening experiment set was performed. This design is specifically intended to screen a large number of potentially important factors that may affect the desired quality characteristic(s). The disadvantage with this design is that, while the main effects of a large number of factors may be determined, knowledge of any non-linear effects is forfeited.

A screening experiment worksheet was generated having 16 experimental runs. The process parameters for the first, second, and fourth composite plies were held constant as follows: placement rate, 2.54 cm/s; compaction load, 1.33 kN; compaction roller temperature, 480°C; torch temperature, 700°C; IR lamp output, 100%. Film tack stage processing parameters held constant were processing speed, 2.54 cm/s; compaction load, 0.56 kN; compaction roller temperature, 360°C. Specimens were peel tested and values for both the peak and average peel strengths were recorded.

The results of the screening experiments are listed in Table 3 in order of their relative importance as determined by the magnitude of their confidence coefficients. Clearly the most significant factors influencing the peel strengths are the melt stage roller temperature, compaction load, and use of the IR lamp during melt staging. It is assumed that the majority of thermal energy available to melt and bond the film to the lower composite ply is transferred via conduction by the heated compaction roller. The relatively minor influence of the torch temperature during the melt stage is again thought to be due to gas flow stagnation in the nip point region. The IR lamp was originally added to the placement machine to serve as a supplemental preheat source due to the inability to elevate the nip point temperatures sufficiently with heated gas alone. The high ranking of the IR heat source is therefore confirmation of the benefit of this additional heat source for material pre-heating. The importance of the compaction load during the film melt stage is thought to be due to an increase in melt flow and interfacial healing at higher compaction pressures.

The processing variables for placement of the upper composite ply were investigated in the study to determine if the presence of the film layer on the substrate would significantly alter the optimal conditions as found for the composite alone. The torch temperature and compaction load were varied for placement of this ply. The torch temperature was found to be more significant during placement of the upper composite ply than during the film melt processing stage. The compaction load for placement of the upper ply was found to be the least important of the screened parameters. This may be explained by the presence of the film layer; the film provides a smooth, resin-rich surface for the upper composite ply to adhere to.

The processing speed during the film melt stage was found to be of borderline importance in the screening experiment. Due to the uncertainty in the significance of this parameter in the process, and to the relatively low values of peel strength obtained in the screening experiments, further experiments designed to optimize the process were carried out at lower placement speeds.

Table 3. Confidence coefficients obtained from process variable screening experiments based on average peel strength data.

Process Variable	Confidence Coefficient
Melt Stage Roller Temperature	0.89
Melt Stage Compaction Load	0.85
Melt Stage IR Lamp	0.80
Melt Stage Processing Speed	0.70
Upper Composite Ply Torch Temperature	0.61
Melt Stage Torch Temperature	0.38
Upper Composite Ply Compaction Load	0.22

2.3.3 Optimization by Design of Experiments and Response Surface Methodology

The results of the Plackett-Burman screening suggest that the three most significant parameters for processing film interleaves, in terms of average peel strength, are the melt stage roller temperature, the melt stage compaction load, and the IR heat source during melt staging. The most insignificant parameters were determined to be the torch temperatures and the compaction load during upper composite ply placement and the torch temperatures during the melt stage. As previously stated, the processing rate during the film melt stage was of borderline importance.

Further investigations to determine the bounds of an optimal process were performed [1]. A series of experiments utilizing a Box-Behnken design were conducted at film melt rates of 0.64 cm/s, 0.95 cm/s, and 1.27 cm/s. Torch temperature values for placement of the upper composite ply and for the melt stage processing pass were held constant at 700°C. The compaction load for placement of the upper composite ply was also held constant at 1.33 kN. The processing rate for all of the composite plies was held constant at 2.54 cm/s. The compaction roller temperature, the compaction load, and the lamp percent output during the film melt stage were varied during the experiments. The melt stage roller temperature range investigated was 400°C to 480°C, the compaction load range was 0.56 kN to 1.22 kN, and the lamp output power range was set from 0% to 100%.

3.0 RESULTS AND DISCUSSION

3.1 Interleaf Processing The peel data obtained from the Box-Behnken optimization experiments of Section 2.3.3 were fitted using a quadratic regression model. The resultant response surfaces were generated using JMP statistical software. An analysis of the results was undertaken to determine the goodness-of-fit for the response surface models at each of the three film melt processing rates. The indicators used to determine the quality of fit of the quadratic model to the data were the R-value, the sum-of-squares of the model (SS_m), and the sum-of-squares of the error (SS_e). An R-value of 1 signifies a perfect fit and complete confidence in the predictive capability of the model. Conversely, low R-values signify a poor fit, and hence an inability of the model to make predictions regarding the dependent variable. Additionally, the goodness-of-fit may be determined from the sum of the squares of the model and the error. Good correlations have SS_e values much less than the SS_m .

The quality of fit of the model for the experiments performed at a melt speed of 0.64 cm/s was found to be the best of the three experiment groups. The R-value for the fit of the experiments performed at this rate was found to be 0.93. The SS_m and the SS_e were found to be 63.75 and 10.02, respectively. It is concluded that a relatively high degree of confidence may be attributed to the model for this data set. The R-value for the model for the experiments performed at 0.95 cm/s melt processing speed was found to be 0.88 and the SS_m and SS_e were found to be 56.38 and 16.62, respectively, indicating justification in the confidence of this model as well. Analysis of the fit of the model for the experiments performed at 1.27 cm/s give an R-value of 0.61 and an SS_m and SS_e of 21.1 and 35.1, respectively. These values would indicate that virtually no confidence can be attributed to the fit of the model for this data set.

Inspection of the response surface plots revealed a general upward trend in peel strength with increases in compaction roller temperature and load at each lamp output setting. An upward trend in maximum strength values with increases in IR lamp output power was observed. Also noted was a general increase in the maximum peel strength as placement rates decreased from 1.27 cm/s to 0.64 cm/s. It may be concluded from the contour plots that the wedge peel strengths are maximized at an IR lamp output of 100% and a placement rate of 0.64 cm/s, conditions which provide the maximum thermal energy flux to the material.

Photomicrographs of the peel specimen having the highest peel strengths at each of the film processing rates are presented in Figures 3, 4, and 5. All three specimens exhibit well-consolidated void-free regions at both the upper and lower film/composite interfaces. A general decrease in film thickness, however, was observed with decreasing film melt processing rates. Measurements of the resultant film thickness for each of the specimens was performed using an optical microscope fitted with a Boeckeler Instruments MicroCode II digital position readout. The microscope magnification power used was 500X. Forty film thickness measurements were recorded for each of the three specimens.



Figure 3. Photomicrograph of 4-ply peel specimen placed at melt stage conditions of 440°C roller temperature, 1.22 kN compaction load and without supplemental IR lamp energy. Shown is specimen having the highest peel strength (8.30 kN/m) of those fabricated at 1.27 cm/s. Average film thickness after processing: 0.0069 cm.

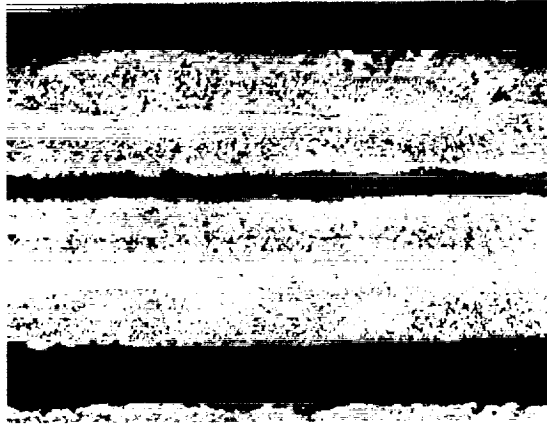


Figure 4. Photomicrograph of 4-ply peel specimen placed at melt stage conditions of 440°C roller temperature, 0.56 kN compaction load, and at 100% lamp power. Shown is specimen having the highest peel strength (8.00 kN/m) of those fabricated at 0.95 cm/s. Average film thickness after processing: 0.0049 cm.

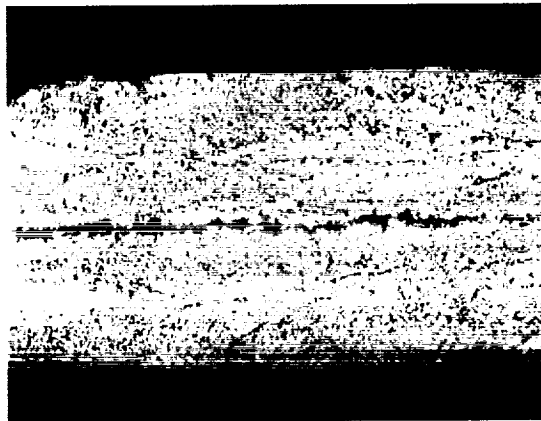


Figure 5. Photomicrograph of 4-ply peel specimen placed at melt stage conditions of 480°C roller temperature, 0.89 kN compaction load and 100% lamp power. Shown is specimen having the highest peel strength (8.60 kN/m) of those fabricated at 0.64 cm/s. Average film thickness after processing: 0.0024cm.

The decrease in film thickness may be primarily attributed to the increased melt flow of the film due to the higher energy fluxes achieved at the lower placement rates. As a result of this increase in melt flow, the amount of resin that adheres to the roller surface also increases. This leads to a 'skimming' of the resin from the specimen during the melt processing stage and to the observed decrease in film thickness. The results of the film thickness measurements are presented in Table 4. Note that the ratio of the standard deviation to the mean thickness increases as film thickness decreases; the thickness of the films processed at higher placement rates is less variable than for those processed at lower rates.

Table 4. Film thickness measurements and standard deviations for the specimens having the highest average peel strengths at each of the melt processing rates.

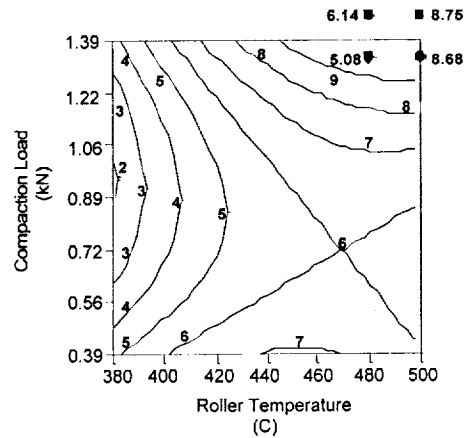
Film Melt Processing Speed (cm/s)	0.64	0.95	1.27
Average Peel Strength (kN/m)	8.60	8.00	8.30
Mean Thickness (cm)	0.0024	0.0049	0.0069
Standard Deviation (cm)	0.0011	0.0018	0.0011
Standard Dev. / Mean Thickness (%)	45.8	36.7	15.9

A final set of experiments was performed in an attempt to confirm the results of the Box-Behnken experiments and to gage the accuracy of the response surface predictions for higher peel strengths. Four-ply peel specimens were fabricated at 100% lamp output power at each of the film melt processing rates. Previous experiments indicated that maximum values of peel strength occurred at high values of roller temperature at each of the three film processing rates. Specimens were therefore fabricated at both 480°C and 500°C melt roller temperatures. Similarly, high values of compaction load were found to produce specimens of higher peel strength. These higher placement settings were used to fabricate additional specimens at each of the three melt processing rates. The response surface for the film melt processing rate of 0.95 cm/s, however, predicted high peel strength values at low compaction force settings. Due to the relatively good fit attributed to this response surface, four specimens were fabricated and tested in this lower compaction load region in addition to the four processed at higher loads. The region of high peel strength for the processing rate of 0.64 cm/s was the largest in size of the three, therefore two additional specimens were fabricated for this experimental set. The results of these experiments are presented graphically in Figure 6. The average peel strength data recorded for each of the test specimens is superimposed onto its respective contour plot.

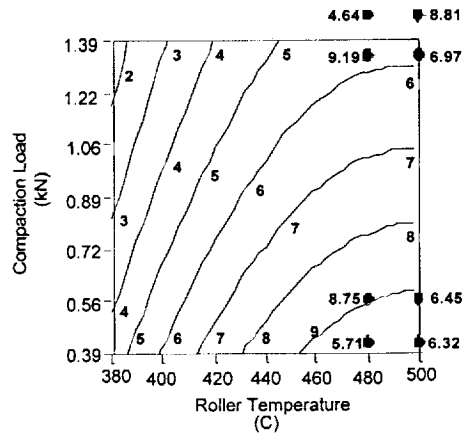
The results for the melt processing rate of 1.27 cm/s show a relatively high degree of variability. This variability may be attributed to the poor fit of the model to the data determined for this processing rate. It should be noted however, that the two specimens fabricated at a roller temperature of 500°C are both significantly higher in peel strength as compared to those placed at 480°C. This would tend to confirm earlier conclusions regarding the importance of conduction heat transfer in the film melt processing stage.

The results for the film processing rate of 0.95 cm/s indicate that peel strength is relatively unaffected by compaction force in the range of roller temperatures investigated. The average peel strength of the specimens fabricated at high compaction load values was 7.04 kN/m, and the average for those placed at lower compaction loads was 6.81 kN/m.

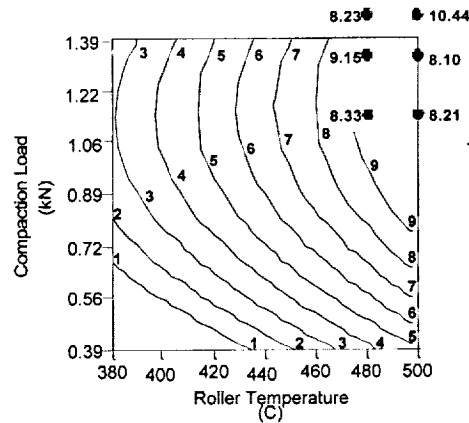
The specimens fabricated at 0.64 cm/s had the highest average peel strength of the three test groups in addition to the smallest variation. The average for this set was 8.74 kN/m. This would appear to be a strong confirmation of both the prediction and the goodness-of-fit of the response surface model for this test set. However minor thermal degradation of the resin was seen to occur during the film melt stage at 0.64 cm/s in some specimens. The roller temperature range used would therefore seem to be very close to an upper bound for the film melt stage pass at this processing rate.



(a) 1.27 cm/s



(b) 0.95 cm/s



(c) 0.64 cm/s

Figure 6. Results of confirmation experiments, (•), superimposed onto response surface plots generated from data of earlier tests. IR lamp output constant at 100%.

3.2 Discussion on the Use and Validity of the Wedge Peel Test Method [6] The wedge peel test initiates and propagates fracture of the specimen at the peel interface in what is essentially opening mode, or Mode I, failure. The ASTM standard test for this failure mode in laminated composite materials is the double-cantilever beam (DCB) test [7]. Several differences between the peel test as performed in this work, and the DCB test should be noted. In the DCB test, the specimen arms are extended by the test frame. This initiates and propagates fracture at the laminate interfaces. During testing, the crack length, load, and specimen arm deflection are recorded for the crack initiation point and for five additional propagation points along the specimen length. Calculations using this data describe the behavior in terms of the amount of energy required to fracture the specimen. In contrast, the wedge peel test is a constant-displacement test; specimen arm displacement is held constant and is fixed by the wedge thickness. Since the crack length is not measured in the peel test, a true fracture energy value is not obtained and therefore a direct one-to-one correspondence to the DCB data cannot be made. An earlier study was performed and results indicated a correlation between the two tests [6].

The motivations for use of the peel test for process development are the simplicity of test specimen geometry, low material usage, and the speed at which testing and data reduction can be performed. The disadvantage of the peel test as performed in the present study is that the influence of the tool/substrate thermal boundary condition is neglected; this boundary condition changes with number of plies placed. A more thorough study in the use of fiber placement for film interleaf processing would attempt to account for this changing parameter [8].

Other research has indicated that increases in interlaminar fracture toughness of from 20% to 45% are possible with interleaved composite systems. A similar comparison was performed on the results of the current study. The five highest values of average peel strength for both the interleaved and non-interleaved peel specimens, regardless of processing conditions, were averaged and compared. The peel strength averages of the non-interleaved and interleaved systems were found to be 7.23 kN/m and 9.27 kN/m, respectively. This represents an increase of 22% for the interleaved materials. This increase is in good agreement with those as previously reported in the literature [9,10]. Additionally, this would further indicate that the wedge peel test and the DCB interlaminar fracture toughness test are likely measuring the same basic failure mechanism.

4.0 CONCLUSIONS

4.1 Summary of Process Development A series of experiments was conducted to determine the optimal processing parameters for the fabrication of composite laminates having interleaf layers by fiber placement. Initial investigations centered on determination of the optimal conditions necessary for processing the composite material without interleaf films. The resulting set of optimal process parameters was then used for the composite for the interleaving process development portion of the study. Preliminary interleaving process trials indicated that for best results, film placement should consist of a two-stage process; a film 'tack' stage to achieve light adhesion of the film to the substrate composite ply, followed by a second 'melt' processing stage to intimately bond the film to the substrate ply. A Plackett-Burman screening experiment was then conducted to determine the relative influence of the process variables on the quality of the

interleaved composite specimen as determined by the wedge peel test method. Processing studies were then focused on determination of the optimal process variable settings found to be of significance in the screening study. The factors having the greatest influence on specimen peel strength were found to be the compaction roller temperature and the compaction load during film melt staging at each of the three placement rates investigated. Summaries of the optimal processing conditions for the composite and composite with interleaves are given in Tables 5 and 6, respectively.

Table 5. Optimal processing parameters for PEEK/IM7 composite.

Process Variable	Value
Lamp Power	100%
Placement Rate	2.54 cm/s
Compaction Force	1.33 kN
Torch Temperature	700°C
Roller Temperature	480°C

Table 6. Optimal processing parameters for PEEK/IM7 composite with PEEK interleaf layer. IR lamp output was 100% for composite plies and for film melt processing stage.

Process Variable	Composite	Film Tack	Film Melt
Torch Temperature (°C)	700	-	700
Roller Temperature (°C)	480	400	480-500
Compaction Load (kN)	1.33	0.22	1.47
Processing Rate (cm/s)	2.54	2.54	0.64

4.2 Suggestions for Future Work The results of the study indicate that the fabrication of composite structures having film interleaf layers by automated fiber placement is possible. The current study brings to light several processing issues that might be addressed in future automated interleaf processing studies.

Initially, plans were in place to investigate the processing of laminates having both 0.0025 cm and 0.0076 cm thick films. After several trials, work with 0.0025 cm films was abandoned as difficulties in achieving complete, high integrity film coverage on the specimen surface were encountered. The ability to have a robust process, one capable of processing very thin films, would be advantageous in that a decrease in overall laminate specific strength accompanies the addition of film interleaf regions within a composite. The results of the present study indicate that a decrease in film thickness is made possible by the process itself. Additional studies might be directed towards controlling the film thickness during processing. This may require close control of such processing variables as compaction roller force and temperature.

Another important processing issue that should be addressed in future work is film gaps and overlaps. Early trials were conducted with film sheets several times the width of the heated compaction roller and composite substrate. It was immediately apparent that, due to local film melting and severe buckling and distortion of the film sheet, film interleaf materials would have to be processed in strip form. Other trials were conducted with films much narrower than the composite substrate in an attempt to determine how accurate (straight) these films could be tacked to the substrate ply. Measurements indicated that film strips could be tacked to within ± 0.026 cm straightness deviation over a length of 46 cm. Accurate preliminary bonding, or tack staging, of the film is critical in terms of integrity of the film layer in the case of gaps, and in uniformity of the film thickness in the case of overlap regions. A substantial increase in film width is seen to occur during film melt stage processing and might conceivably eliminate any gaps in a completed film layer. Perhaps a more conservative approach might be to study the effect of the overlapping of films during the tack stage; relying on the melt stage process to eliminate any gaps would require a high level of confidence in the predictability of the extent of film melt flow.

5.0 REFERENCES

1. Hulcher, A.B., M.S. Thesis, Old Dominion University, Norfolk, VA, 2000.
2. Munjal, A.K., 1987. Advanced Composites III: Expanding the Technology, ASM, Metals Park, Ohio, pp. 53-56.
3. Goetz, R., Ryan, R., Whitaker, A.F., "Final Report of the X-33 Liquid Hydrogen Tank Test Investigation Team", NASA/Independent Review Report, May 2000.
4. Towell, T.W., Johnston, N.J., Grenoble, R.W., Marchello, J.M., Cox, W.R., Science of Advanced Materials and Processes Engineering Series, 41:1701-1711.
5. Grenoble, R.W., M.S. Thesis, Old Dominion University, Norfolk, VA, 1998.
6. Hulcher, A.B., Marchello, J.M., and Hinkley, J.A., 1999. Journal of Advanced Materials, 31(3):37-43.
7. ASTM D 5528. 1994. Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites, 14.02:272-281.
8. Pitchumani, R., Gillespie, J.W., and Lamontia, M.A., 1997. Journal of Composite Materials, 31(3):244-275.
9. Dolan, G.L., Masters, J.E., 1998. International SAMPE Technical Conference Series, 20:34-45.
10. Hirschbuehler, K.R., 1985. Toughened Composites, ASTM, pp.61-73.

ACKNOWLEDGEMENTS

The authors would like to express their thanks to the following individuals without whose help this work would not have been possible: Harry L. Belvin, Pert Razon and John Kirtley of the NASA Langley Research Center.